



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification³ : B25D 27/04	A1	(11) International Publication Number: WO 85/ 01238 (43) International Publication Date: 28 March 1985 (28.03.85)
(21) International Application Number: PCT/SE84/00287 (22) International Filing Date: 31 August 1984 (31.08.84) (31) Priority Application Number: 8304872-8 (32) Priority Date: 12 September 1983 (12.09.83) (33) Priority Country: SE (71) Applicant (for all designated States except US): AB ROVAC [SE/SE]; Box 4048, S-511 04 Kinna (SE). (72) Inventor; and (75) Inventor/Applicant (for US only) : BAKKELUNN, Terje [NO/SE]; Linnegatan 45, S-413 08 Göteborg (SE). (74) Agent: KARLSSON, Berne; Box 2078, S-137 02 Västerhaninge (SE).		(81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), FR (European patent), GB (European patent), JP, LU (European patent), NL (European patent), SE (European patent), US. Published <i>With international search report.</i> <i>In English translation (filed in Swedish).</i>
(54) Title: METHOD FOR THE MANUFACTURE OF PRODUCT COMPRISING FOAMED PLASTIC MATERIAL AND USING A LOW-PRESSURE CHAMBER		
(57) Abstract A propellant is mixed with unsaturated polyester inside a first space (A) at a first pressure, for example at the atmospheric pressure. The polyester is then sprayed on the whole or parts of an open or exposed mould which is applied in a low-pressure chamber having a powerful negative pressure of 60-95% vacuum. In order to spray a first layer, polyester having a first amount of propellant is sprayed on the mould. At the initiation the propellant causes a powerful foaming effect at which bubbles are created and/or expanded in the polyester. In order to spray a second layer, which can be applied before or after the first layer, polyester having a second amount of propellant, or is without propellant, is sprayed on the mould, which causes fewer bubbles or no bubbles at all. The resulting polyester which comprises formed and expanded bubbles is bound (is allowed to take rigid shape/is polymerized) by the use of a short tack-free time in the low pressure, said short time being preferably 5-10 minutes.		

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TITLE

Method for the manufacture of product comprising foamed plastic material and using a low-pressure chamber.

TECHNICAL FIELD

5 The present invention relates to a method for the manufacture of a product comprising foamed plastic material in a first layer. The product comprises a second layer as well. The second layer is connected to the first layer and having higher density than the first layer.

10 PRIOR ART

It is usual in the manufacture of foamed plastic products to utilize some form of chemical propellant which is dissolved in the base material or is added to it immediately before the foaming process is to commence. The sulphone hydrazides may be mentioned as an example
15 of such a propellant. The foaming process can also be brought about by the use of physical propellants in the form of a volatile gas such as freon (for example freon 11) which is dissolved in (that is to say forms part of a single-phase system with) the base material. A familiar example of the last-mentioned method is provided by the
20 manufacture of rigid polyurethane foam. The use of carbon dioxide as a propellant has also been disclosed.

Activation of the propellant in question, for example by means of catalysts, heat and/or pressure, will cause the propellant to generate gas, resulting in the formation of bubbles which are
25 apparent in the finished product. The familiar methods used until now for the manufacture of products falling within this category nevertheless call for the use of a comparatively large quantity of propellant of up to 10 per cent by weight or more. The use of such a large quantity of foaming additives will result in various
30 manufacturing problems, for example problems associated with curing and storage which, when viewed as a whole, complicate the manufacturing process and impair the product. Furthermore, the choice of base materials and the range of possible variations in the



production conditions, for example the variations in temperature, are restricted to a certain extent, which has a negative effect on the result.

DESCRIPTION OF INVENTION

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Technical Problem

A wish associated with foamed plastic products falling within this category is for manufacturing processes to be available which provide the conditions necessary for a major reduction to be achieved in the quantity of the foaming additives used. Said additives have
10 tendencies to disturb the curing process and cause cracks. Demands on durable and cheap products are available as well.

Further wishes are to be able to allot, if necessary, the different layers of the product different thicknesses by a simple control procedure. There is also a wish to be able to provide the product
15 with exact positioned reinforcements, if necessary.

THE SOLUTION

The present invention proposes a method for the manufacture of a product having a foamed plastic material which will resolve, amongst other things, the problem outlined above on the basis of the usage of
20 unsaturated polyester and that considerably increased foaming effect can be achieved from the use of a propellant at a high degree of low pressure, with a vacuum of 60-95% being envisaged in this case. The essential characteristics of the new method are, amongst other things, that inside a first space, which is at a first pressure, the
25 aforementioned propellant is mixed with or dissolved in a curing agent-compatible unsaturated polyester. Second feature of the invention comprises the usage of a second space in the form of a negative pressure chamber which is at that high degree of negative pressure of 60-95% vacuum. The last mentioned pressure is quite below
30 the first pressure. Said chamber has an open or exposed mould and on this one the mixed polyester is sprayed. In order to attain the first layer, polyester mixed with a first amount of propellant is sprayed on the whole or parts of the mould in said low pressure. The foaming



effect is initiated by means of a curing agent, initiator(-s) and/or said negative pressures and the propellant causes an powerful foaming effect in which bubbles are created and/or expanded in the sprayed polyester. In order to attain the second layer, polyester having a second amount propellant, or not any propellant at all, is sprayed on the whole or parts of the mould. Then fewer bubbles than in the case with the first amount, or not any bubbles at all, are created and/or expanded in the second polyester layer. Said first layer is sprayed on the mould before said second layer, or vice versa. Said created and/or expanded bubbles, in at least the first layer, are bound in the polyester by allowing this one to take rigid shape/to be polymerized in the negative pressure for a short time, for example 30 seconds - 20 minutes, preferable 2-10 minutes.

In an embodiment it is proposed that carbon dioxide is used as the propellant. Furthermore, the spacer material in certain embodiments is reinforced with carbon fibres or glass fibres. The spacer material can be provided in a preferred sandwich construction with a covering layer of higher density on each side of the foamed core. Designs having several layers of foamed or not/less foamed polyester are simple to manufacture (control) according to the invention.

In further developments of the idea of invention it is proposed that measures shall be adopted in order to prevent the situation arising in this context in which an excessively rapid rate of ascent is achieved by the gas bubbles produced at the first pressure.

Accordingly, the base material must, for example, be essentially free from wetting agents. Also, the use of small bubbles is preferable in order to reduce the rate of ascent. The material selected will preferably be a thixotropic polyester having a viscosity at high shear rates of preferably 140-300 mPa.s. The use of a curing agent system providing a short curing time is also recommended. Bubbles are able to be created in the polyester in the first space. Said bubbles are expanded in the low-pressure, together with newly created bubbles, if any, in the low pressure.

It is preferable to utilize for the formation and the expansion of the gas bubbles a low-pressure chamber capable of operating at a vacuum of 60-95%, and preferably 70-90%. A chamber of this type can include a suitable space for the product and can be equipped with,



amongst other things, openable and closable hatches and, if appropriate, material application organs and air evacuation equipment, etc. The gas bubbles are formed either from gas, for example carbon dioxide, dissolved in the material, or by causing gas to be driven off in conjunction with the dissociation of a chemical propellant. The different layers can be sprayed wet in wet or with certain time intervals where the preceding layer takes a more or less rigid shape before the application of the next layer.

ADVANTAGES

By means of the invention it is possible to obtain a product having a foamed layer which comprises a significantly reduced amount of chemical foaming additives or physical propellants dissolved in the base material. It is worth mentioning in this respect that it is possible, by the use of a 85-90% vacuum, to reduce the quantity of propellant to 1/8 of that encountered in commercially available foamed products of equivalent kind. In this way products which exhibit high mechanical characteristics (less dependence of strength) can be obtained at low cost. The working environment can be designed to a high standard. The density and/or the reinforcement of the finished product is easily controlled by the equipment which is used. The curing of the product may be made more simple and more distinct, thereby providing the necessary conditions for high-quality products. Each second layer can be made reinforced and not foamed. Only certain parts of the mould can be provided with foamed polyester while other parts can be provided with second layers, which easily can be made for attaining reinforced portions. The polyester proposes conditions for stronger products and the new method solves the problems of the low gel temperature of polyester. Low bubble pressure is attained which proposes possibility for crack free curing process.

FIGURES

Preferred embodiments of a method for the manufacture of two-layers product is described below by means of four examples, with simultaneous reference to the accompanying drawings, in which:

Figure 1 illustrates the principle behind the manufacture of a spacer material in accordance with the first example; and



Figure 2 illustrates the principle behind the manufacture of a spacer material in accordance with the second example; and

Figure 3 illustrates examples of a sandwich construction.

5 THE BEST EMBODIMENT

Example 1 (Figure 1)

In a container 1 is placed unsaturated polyester (suitable for spray-up operation), for example laminating polyester of the type used in the small boat industry. The polyester is thixotropic and exhibits a viscosity at high shear rates of, for example 150 mpa.s. The polyester should preferably be at least essentially free from the wetting agents which are otherwise normally used to facilitate the admixture of the filler. The unsaturated polyester should preferably be modified in such a way that rapid binding can be achieved after the addition of the curing agents, for example peroxides. The curing time or the gel time should preferably be selected within the range 30 seconds - 20 minutes, and preferably 2 - 10 minutes. The curing time can be adjusted in a previously disclosed fashion by means of accelerators, inhibitors and curing agents.

20

A slowly-rotating agitator 2, for example an agitator rotating at a speed of about 15-50 r/min, is utilized for stirring in conjunction with the admixture of a chemical propellant, for example a sulphone hydrazide, or a physical propellant which may be in the form of FREON 11. The quantity of propellant used in the case of a chemical propellant is about 0.2 - 1.0, and preferably about 0.5 per cent by weight of the total mixture of polyester and propellant. (Under normal circumstances using a previously disclosed method 3-5 per cent by weight of the same propellant would be needed). The corresponding values in the case of physical propellants are about 0.05 - 0.5, and preferably about 0.1 per cent by weight of the total mixture of polyester and propellant. The admixture of the propellant is carried out in a first space A having a first pressure which essentially correspond to the atmospheric pressure or is higher than the last mentioned pressure.

30



After the admixture of the propellant the material is pumped by means of a pump 3 to a material application device 4, for example a spray pistol of a previously known type. A similarly previously known cutter 5, for example a fibre glass cutter, may be mounted on the pistol. With the help of the cutter chopped fibres 6 are thrown into the jet of polyester 7 which is directed towards an open or exposed mould F. Curing agents, for example peroxides, for the polyester are added from a container B and are mixed in the pistol 4 with the polyester in a previously known fashion. The curing agent may alternatively be mixed with the jet outside the pistol.

In the case of non-reinforced products the cutter is not activated or may be omitted.

Spraying on the mould takes place in a second space 8 which can comprise a space 9 inside a low-pressure chamber 10 in accordance with Figure 1. The chamber is provided with material application (spray) organs and, if appropriate, with cutter equipment corresponding to those illustrated. Said application organs can be adapted on a not shown robot which is automatically controlled. The application can be made on the whole or parts of the mould and with different thicknesses, distribution, etc., of polyester, reinforcements, etc.

Alternatively the entire process, that is to say including the agitation operation, may take place inside the chamber 10, the space 9 of which is in that case kept at the first pressure during agitation and during the addition of the propellant. The curing agent may be added during the agitation operation, during addition to the mould and/or after the chamber has been actuated so as to provide low pressure.

The chamber in dependence of actuation of the air evacuation equipment will in this case be of the previously disclosed kind which is able to produce a high degree of low pressure at 60-95%, and preferably 70-90% vacuum. In the event of a chemical propellant being used, this is activated to give off gas preferably by means of the curing agent. The low pressure permits a higher degree of expansion of the gas bubbles to take place than is possible at atmospheric



pressure. In the event of a physical propellant being used, this is activated by the low pressure, which also permits a lower density to be achieved in the finished foam than is possible at atmospheric pressure for an identical quantity of propellant. The short curing time or gel time of 30 seconds - 20 minutes, and preferably of 2 - 10 minutes for the material results in the formed and expanded bubbles being bound in the material in a rapid and effective manner. The application of the plastics material and the curing agent may take place at low pressure. The admixture of the propellant always takes place essentially at atmospheric pressure or a pressure above (for example 40-60 bar according to below) the same. The curing of the spacer material takes place at low pressure.

Products of least two layers can be made. The first layer is foamed and the second layer is less foamed than the first layer or having a higher density. The choice of porosity is controlled by means of the amount propellant, the negative pressure and the polyester type. The layers are applied preferably wet in wet.

In the event of reinforcement of the product being required, this can be achieved with relatively very good characteristics. For the first layer the following is valid. The admixture of, for example, 30 per cent by weight of chopped fibres (glass fibre) at a density of about 800 kg/m^3 (which must be compared with a density of about 1500 kg/m^3 for non-foamed products) will result in a bending strength of about 22 mPa, a modulus of elasticity on bending of about 1000 mPa, and a compressive strength of about 14 mPa. The finished material may also be used for sandwich constructions having two or more layers. It may be mentioned from a purely general point of view that the degree of reinforcement of the first layer can be selected within a range of 10-40 per cent by weight, and preferably 20-30 per cent by weight.

Example 2 (Figure 2)

A propellant gas other than FREON 11, for example carbon dioxide, may be used and is applied at a pressure such that the carbon dioxide will be dissolved either in full or in part in the polyester or equivalent substance. The polyester may be of the same quality



as above. In this case polyester is pumped by means of a pump 16 from the container 1' to a material application organ, for example a spray pistol 13. In the pipe 14 between the pump and the spray pistol are so arranged as to be capable of being connected a source 15 of carbon dioxide (a cylinder) and a mixing organ 16 (a so-called static mixer) which mixes the carbon dioxide into the polyester. The source 15 operates preferably at about 40-60 bar. From the spray pistol 13 the material is applied on the exoosed mould at the powerful negative pressure according to above, e.g. in the aforementioned low-pressure chamber. The addition of the curing agent and of any reinforcement which may be required can take place either at the first pressure or at the high degree of low pressure produced by the chamber and which is of the order or magnitude indicated above. The curing time will be short; see above.

Figure 3 illustrates examples of a sandwich construction in which the spacer material (the core) is identified by the reference designation 18, the covering layer by 19 and 20, and the expanded bubbles of the spacer material by 21. The fibre reinforcement is identified by the reference designation 22. The porosity is controlled by the amount of applied carbon dioxide.

Example 3

Polyester and a chemical propellant are mixed together inside a container in accordance with the above my menas of a slowly-rotating agitator and at atmospheric pressure. The resulting mixture is applied on a mould together with a curing agent, for example organic peroxide and, if necessary, glass fibre, at a low pressure of 70-90% of vacuum, for example. Seeing to the first layer, 0.5 per cent by weight of propellant at about 85% of vacuum will produce a similar foaming effect to that produced by 2 per cent by weight of propellant at atmospheric pressure, not including reinforcement in both cases.

The following components may be used. Polyester in the form A360/246A supplied by BP Chemicals. Propellant in the form Cellogen XP-100 supplied by Uniroyal Chemicals. Peroxide in the form Butanox M-50 supplied by AKZO Chemicals. The amout of peroxide used should be 2 per cent by weight. For each second layer the propellant is reduced or cancelled.



What will be achieved for the first layer, apart from a reduced consumption of the propellant, is more reliable curing and a lower temperature during curing, resulting in less of a tendency to crack formation, and in more even curing, etc. The negative pressure results in lower bubble pressure in the curing procedure.

Example 4

Polyester and a chemical propellant are mixed together and are applied with a and, if necessary, reinforcement in a form in accordance with Example 3. The polyester in this case is in the form Leguval SF 38 supplied by Bayer AG. The propellant is LE supplied by Bayer AG, and the peroxide (2 per cent by weight) is Butanox M-50 supplied by AKZO Chemicals. Tests for the first layer have shown that the use of 2 per cent by weight of the propellant at atmospheric pressure will provide a density of 1.1 g/cm^3 at 2 per cent by weight and at 90% vacuum the density became 0.4 g/cm^3 , that is to say a lower density can be achieved, or else a considerably reduced propellant consumption can be achieved for a given density.

In the case of Examples 3 and 4 the admixture of fillers of a previously disclosed kind is permissible in order to provide, for example, fire-resistance, an even cheaper finished product, and the suppression of high temperatures during curing, etc. The use of glass fibre reinforcement has no significant effect on foaming. The invention is not restricted to the embodiments illustrated above by way of example, but may undergo modifications within the context of the following Patent Claims and the idea of invention.

PATENT CLAIMS

1. Method for the manufacture of a product comprising foamed plastic material in a first layer and a second layer adapted to the first layer and having higher density than the first layer,
5 c h a r a c t e r i z e d in that a propellant is mixed together with unsaturated polyester in a first space which is at a first pressure, in that in a second space in the form of a negative pressure chamber which is at that high degree of negative pressure of 60-95% vacuum and in such a way is essentially below the first pressure, is sprayed
10 on the whole or parts of an exposed mould:

a) in order to attain the first layer, polyester mixed together with a first amount of propellant, which by initiation by means of a curing agent, initiator(-s) and/or said negative pressure causes a powerful foaming effect, whereby bubbles are created and/or
15 expanded in the sprayed polyester,
b) in order to attain the second layer, polyester mixed together with a second amount propellant which is less than said first amount propellant, or without any propellant at all, whereby fewer bubbles than in the case of a), or not any bubbles at all, are
20 created and/or expanded in the second polyester layer,
whereby the step of point a) is performed before the step of point b), or vice versa, and in that the created and/or expanded bubbles in at least the first layer are bound in the polyester by allowing this one to take rigid shape/to be polymerized in the negative pressure
25 for a short time, for example 30 seconds - 20 minutes, preferable 2-10 minutes.

2. Method in accordance with Patent Claim 1, c h a r a c t e r i z e d in that inside a container the propellant is mixed with the polyester by means of a slowly-rotating agitator (2), for example an agitator
30 rotating at a speed of 15-50 r/min, in that the polyester with the propellant mixed in is pumped by means of a pump (3) to a material application device, for example a spray pistol, and in that a curing agent is added to the jet of the polyester (7) issuing from the material application device or is mixed with the polyester inside the
35 device (4).



3. Method in accordance with Patent Claim 1, characterized in that the polyester is pumped by means of a pump (12) to a material application organ (13), in that between the pump and the material application organ is introduced a propellant, for example carbon dioxide, from a pressure source (15) which together with mixing organs (16) will cause the propellant to be mixed into the polyester.

4. Method in accordance with Patent Claim 3, characterized in that the curing agent is added separately in relation to the material application organ at atmospheric pressure or at the aforementioned low pressure.

5. Method in accordance with any of the Patent Claims 1-4, characterized in that the reinforcement material (6), preferably in the form of chopped carbon fibres and/or glass fibres, is mixed into the polyester in conjunction with its addition to the mould.

6. Method in accordance with Patent Claim 5, characterized in that the reinforcement material is added at a rate of between 10-40 per cent by weight, and preferably 20-30 per cent by weight.

7. Method in accordance with any of the foregoing Patent Claims, characterized in that a thixotropic and preferably unsaturated polyester, for example a laminating polyester, is added to the container in question inside which the admixture of the propellant takes place, and in that a curing agent in the form of peroxide is added to the polyester.

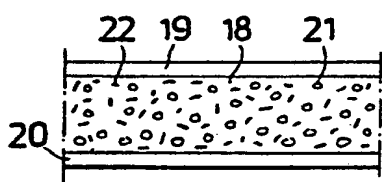
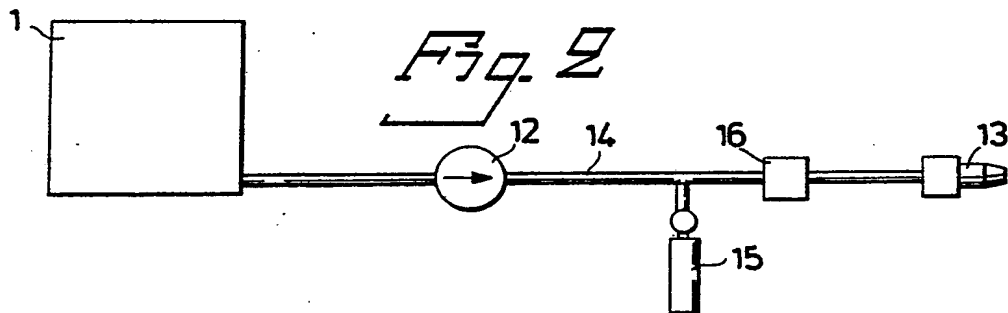
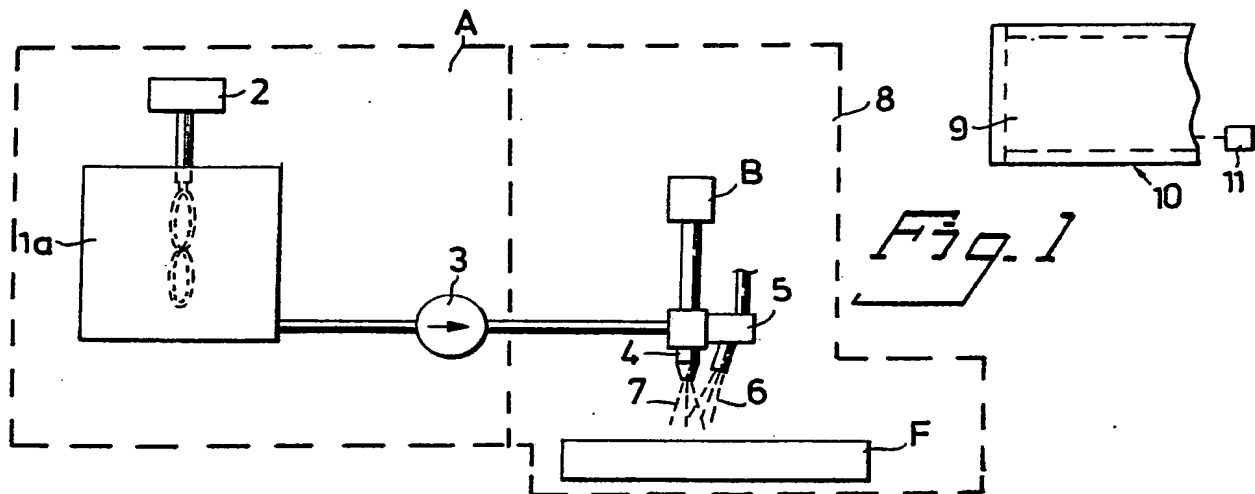
8. Method in accordance with any of the Patent Claims 1-4 and 7, characterized in that the quantity of propellant is selected so that the density of non-reinforced foamed polyester will be 170-600 kg/m³.



9. Method in accordance with any of the foregoing Patent Claims,
c h a r a c t e r i z e d in that the first layer is provided with two second
layer, one second layer on each side, and in that each second layer
is adapted to two first layers if more than one first layer exists,
5 and so on.

10. Method in accordance with any of the foregoing Patent Claims,
c h a r a c t e r i z e d in that the different layers are sprayed wet in
wet.





INTERNATIONAL SEARCH REPORT

International Application No PCT/SE84/00287

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ¹ According to International Patent Classification (IPC) or to both National Classification and IPC ³ <div style="text-align: center; font-family: monospace; font-size: 1.2em;">B 25 D 27/04</div>						
II. FIELDS SEARCHED <div style="text-align: center; font-size: 0.8em;">Minimum Documentation Searched ⁴</div> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 25%; font-size: 0.8em;">Classification System</th> <th style="font-size: 0.8em;">Classification Symbols</th> </tr> <tr> <td style="padding: 5px;">IPC 3 US C1</td> <td style="padding: 5px;">B 25 D 27/00, 02, 04; B 32 B 5/18, 20, 27/36 264:41, 45, 45.3, 50, 51; 425:4; 428:315, 317</td> </tr> </table> <div style="text-align: center; font-size: 0.8em; margin-top: 5px;">Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁵</div>			Classification System	Classification Symbols	IPC 3 US C1	B 25 D 27/00, 02, 04; B 32 B 5/18, 20, 27/36 264:41, 45, 45.3, 50, 51; 425:4; 428:315, 317
Classification System	Classification Symbols					
IPC 3 US C1	B 25 D 27/00, 02, 04; B 32 B 5/18, 20, 27/36 264:41, 45, 45.3, 50, 51; 425:4; 428:315, 317					
<div style="text-align: center; font-family: monospace; font-size: 1.2em;">SE, NO, DK, FI classes as above</div>						
III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴						
Category ⁶	Citation of Document, ¹⁴ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸				
A	SE, B, 416 118 (BRITISH INDUSTRIAL PLASTICS LTD) 1 December 1980					
A	FR, A, 2 458 381 (INDUSTRIES ET TECHNIQUES D'AMENLEMENT) 2 January 1981					
A	FR, A, 2 413 198 (S.A. STRATIFORME) 27 July 1979					
A	NO, B, 135 308 (ACRO POLYMERS INC) 13 December 1976					
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>¹⁵ Special categories of cited documents: ¹⁵</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p> </div> </div>						
IV. CERTIFICATION						
Date of the Actual Completion of the International Search ¹ <div style="text-align: center; font-family: monospace; font-size: 1.2em;">1984-11-09</div>	Date of Mailing of this International Search Report ¹ <div style="text-align: center; font-family: monospace; font-size: 1.2em;">1984-11-13</div>					
International Searching Authority ¹ <div style="text-align: center; font-family: monospace; font-size: 1.2em;">Swedish Patent Office</div>	Signature of Authorized Officer ¹⁰ <div style="text-align: center;"> <div style="text-align: center; font-family: monospace; font-size: 1.2em;">C. Westberg</div> </div>					